

A FURNACE FOR HIGH TEMPERATURE POWDER DIFFRACTOMETRY

HIGH temperature attachment for powder diffractometer is a very useful device for studying several important properties of crystalline materials. A large variety of heating devices have already been described in literature¹. In this letter we present the design and function of a furnace for use upto 700° C on a Philips powder diffractometer. The salient features of this furnace are: (i) the heater assembly can be easily attached or detached to the sample holder and (ii) the simple cooling device which maintains the diffractometer arm at 30° C when the sample is heated at 700° C.

The cross-sectional view of the furnace fixed to the conventional sample holder used with the Philips diffractometer is shown in Fig. 1. The heating device is fixed below the central opening (a) where the sample under study is kept. The furnace assembly consists of a small porcelain trough (b) 35 × 15 × 8.5 mm in size, the bottom of which is filled with layers of alumina (c) and mica sheets (d). The heating element consists of a 5" long, 245 WG nichrome wire (e) wound in a ziz-zag fashion and is fitted on the mica sheets (d). The heater leads are taken downward through two vertical side holes (f) made along the edges of the porcelain trough and fused to copper wires connected to the power supply. The filament wire is further covered by a thin layer of mica sheet. The sample is mounted in contact with the mica sheet which provides electrical insulation between the filament wire and the sample. This furnace assembly, as such, could be fixed to the sample holder by means of four corner screws (g).

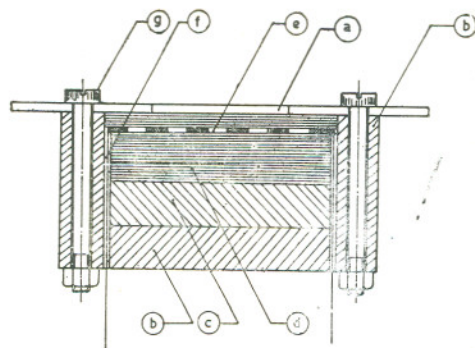


FIG. 1. Cross-sectional view of the furnace fixed to the sample holder.

The filament wire is heated by stabilised a.c. voltage supplied through a dimmerstat. Fine current control is achieved by using two current-sensitive step-down transformers. With this simple circuit it was possible to control the temperature to an accuracy of $\pm 5^\circ \text{C}$. In order to prevent the diffractometer arm (h) from getting heated, two 10 cm long copper tubes (i) (1.5 mm i.d.) are suitably bent and brazed to the lower as well as the upper sides of the brass sample holder. Water which is continuously circulated through the copper tubes linked by a short rubber tubing (j) act as the coolant during high temperature experiments. The positioning of the copper tubes is such that they do not hinder the fixing of the sample holder to the diffractometer shaft and they do not also intercept the x-ray beam. With a rate of flow of 350 ml of water per minute, the diffractometer arm could safely be maintained at 30° C when the sample was heated to 700° C. The temperature of the sample is measured by means of a chromel-alumel thermocouple (k) which is inserted through a slightly bent metallic strip (l) fixed to the top surface of the sample holder. In order to check for thermal gradients along the surface of the sample, the thermocouple could easily be moved under the metallic strip and could be fixed in any position. The output of the thermocouple is fed directly to a digital millivoltmeter which reads the thermal e.m.f. to an accuracy of 0.1 millivolt. At 700° C, the temperature was found to be stable within $\pm 5^\circ \text{C}$ for a period of about three hours, with a power consumption of about 75 watts. As the sample is fixed directly above the mica-

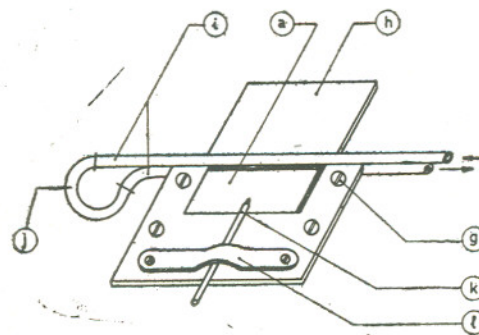


FIG. 2. Cooling and thermocouple arrangement.

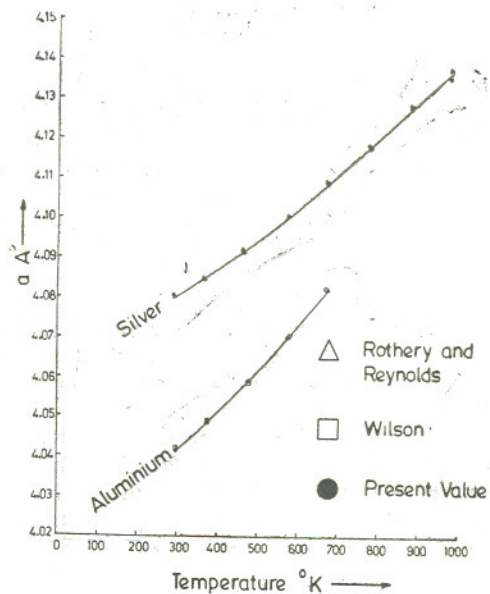


FIG. 3. Temperature variation of lattice parameters of aluminium and silver.

covered filament wire, no appreciable temperature gradient was observed along its surface.

The performance of the heater was tested by measuring the lattice parameters of polycrystalline aluminium upto 400° C and silver upto 700° C. The values plotted in Fig. 3 are found to compare well with those reported in literature^{2,3}.

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1. McKinstry, H. A., *J. Appl. Phys.*, 1970, 41, 5074.
2. Wilson, A. J. C., *Proc. Phys. Soc. (Lond)*, 1941, 53, 235.
3. Hume Rothery, W. and Reynolds, P. W., *Proc. Roy. Soc.*, 1938, 167, 25.